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Microstructural Characterization of Excel Zirconium Alloy

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Abstract

Excel zirconium alloy (Zr-3.5Sn-0.8Mo-0.8Nb) was manufactured in the 80's within the research programs for the development of high strength materials for CANDU (CANada Deuterium Uranium) pressure tubes. Excel test results showed a better dimensional stability, a higher creep resistance and its hydrides are less susceptible to reorientation during service, compared with the current material in use, Zr-2.5Nb. These factors extend the useful life of the tubes and make Excel a good option for the production of pressure tubes. The aim of the present work is to characterize the microstructure of this alloy analyzing specimens with different contents of hydrogen. The phases were studied using optical microscopy, SEM, TEM and X-ray diffraction. The quantitative chemical analysis was performed using EDS technique.

*Keywords:*Zirconium alloys; Excel alloy; pressure tube; microstructure; hydrides.

1. Introduction

Zirconium alloys are used in nuclear applications due to the combination of good mechanical properties such as high strength and creep resistance, reasonable resistance to corrosion and low neutron capture cross-section.

The design of Generation IV CANDU reactors requires higher creep resistant alloys than Zr-2.5%Nb which is currently used for the production of pressure tubes. For this purpose Excel alloy (Zr-3.5%Sn-0.8%Mo-0.8%Nb) was developed in the 80's by AECL Chalk River Nuclear Laboratories and the tests performed showed a higher tensile

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strength, a lower creep deformation and a higher dimensional stability during operation (Cheadle et al., 1980; Ibrahim et al., 1985).

A very important parameter in the safety of the pressure tubes is the presence of zirconium hydrides. These brittle platelets can affect the tube tolerance to longitudinal defects during a reactor shut down. Under certain conditions of stress, temperature and hydrogen concentration, the component is susceptible to failure through the mechanism of delayed hydride cracking (DHC). Excel alloy exhibits higher resistance to hydride reorientation under stress, which make more difficult crack initiation and the subsequent propagation through DHC (Ells et al., 1995).These comparative advantages make Excel a good candidate to replace Zr-2.5Nb alloy. Therefore, it is important to study and characterize the microstructure of this particular zirconium alloy, which allows the prediction of the material performance in nuclear applications.

In the present work the microstructure of Excel samples has been studied by optical, scanning (SEM) and transmission electron microscopy (TEM) techniques. The phase identification and the textural effects in the samples were performed using X-ray diffraction (XRD). The chemical composition was measured by energy dispersive X-ray spectroscopy (EDS).

2. Experimental method

The material used in this work came from an Excel pressure tube manufactured by AECL (Cheadle et al., 1984). The chemical composition is shown in Table 1.

Table 1. Chemical composition of the studied Excel pressure tube.

Tube N°	Sn (*wt%*)	Mo (*wt%*)	Nb (*wt%*)	O (*ppm*)	H (*ppm*)	Zr
254 Front	3.38	0.78	0.82	1118	54	Balance
254 Back	3.47	0.81	0.80	1115	38	Balance

Excel pressure tube was fabricated according to CANDU specifications (Cheadle et al., 1980), by hot extrusion at a ratio of 13.5:1 after preheating to 760°C, followed by 5% cold drawing. The last stage consisted of a stress relief treatment at 400°C for 24 hours.

The samples analyzed in this study were taken from specimens with different hydrogen concentrations, provided by Hydrogen in Materials Division of CNEA. Specimens were cut into radial-circumferential (RC) and radial-axial (RA) sections and the obtained samples were initially studied by optical and scanning electron microscopy (SEM) techniques. For this purpose, the samples were ground with SiC papers followed by chemical polishing using a solution of 48% lactic acid, 48% HNO_3 and 4% HF, to reveal the hydrides and the present phases in the material.

The SEM micrographs were taken using a FEI Quanta 200 microscope operating in the secondary electron mode at 25 kV and a working distance between 8 and 14 mm. The image analysis was done using the Scandium SEM software and the hydrides size was measured and recorded.

X-ray diffraction (XRD) tests were performed in a Philips PW 3710 diffractometer operating at 40 kV with copper anode without monochromator. The step size used was 0.026 and the time per step was 250 seconds. The massive samples selected for XRD were taken from the as-received pressure tube material (0ppm of hydrogen charge) and from specimens with 64 and 122ppm of hydrogen.

The samples for transmission electron microscopy (TEM) were cut into thin slices followed by hand grinding on P600 SiC paper to a thickness of 0,15mm. After that, the foils were punch into 3 mm disks which were then thinned electro-chemically using a solution of 90% ethanol and 10% perchloric acid at -45ºC in a Tenupol-5 twin-jet electropolisher.

Transmission electron microscopy (TEM) and energy dispersive X-ray spectroscopy (EDS) analysis were carried out in a Philips CM 200 equipped with an EDAX detector. The operating conditions for EDS study were an accelerating voltage of 160 kV and a beam size between 25 to 35 nm for all the samples. The quantitative chemical analysis was done using the TEM Genesis software. The KAB factor was selected according to the Mott Massey theoretical model applying a Gaussian fit.